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Just the Facts...

Matrix Spikes for Environmental Sample Analysis – How They are Used and Customer Options

Matrix Spikes- What are They?

Matrix spike (MS) samples are quality control (QC) samples employed to evaluate the effect a particular sample matrix has on the accuracy of a measurement. A matrix spike duplicate (MSD) sample is theoretically equal to the corresponding matrix spike sample and provides a means of measuring method precision. Although the MS and MSD recoveries do provide information on the overall efficiency of the analytical methods used, true method efficiency is better evaluated with laboratory control samples (LCS), which are required by most methods. The LCS is either a 'clean' matrix, free of interferences, or a matrix specifically addressed by the particular method (which makes the LCS recovery a better indicator of the efficiency of the method itself).

Analysis of MS and/or MSD samples is required by many of the environmental laboratory methods performed in the Directorate of Laboratory Sciences (DLS). A matrix spike or matrix spike duplicate QC sample is prepared by adding a known amount of the target analyte(s) to an aliquot of the field sample. These aliquots are separate from (but theoretically equal to) the aliquot to be used to report the target analyte concentrations in the sample. The recovery of the matrix spike is calculated using the following formula:

$$\frac{A_{ms} - A_{fs}}{A_a} \times 100$$

where: A_{ms} = the amount of target analyte measured in the matrix spike sample
A_{fs} = the amount of target analyte measured in the corresponding field sample
A_a = the amount of target analyte spiked (into the matrix spike sample)

The recovery of a matrix spike provides an indication of how efficient the analytical procedure (sample preparation, if applicable, and analysis) was <u>for the particular sample/sample matrix</u> used for the matrix spike. If the matrix spike recovery does not fall within the method acceptance criteria, it may be an indication of sample matrix interferences. However, the matrix interference may only be present in the sample used for the matrix spike. When evaluating MS and MSD recoveries, data users should keep these two points in mind.

1. The suggestion of the presence or absence of matrix interference in a particular sample (as suggested by the MS recovery) should not necessarily be applied to other samples from a particular site or particular analytical batch. The data user may choose to apply the MS/MSD information (whether or not matrix interference may be present) from one sample to a group (or batch) of samples only if there is specific knowledge that the group of samples have a matrix similar to that of the MS/MSD sample.

Environmental Health Consultant
U.S. Army Center for Health Promotion and Preventive Medicine
Aberdeen Proving Ground, MD 21010-5403
410-436-8398 or DSN 584-8398

2. Most analytical methods require that MS samples (and possibly MSD samples) be inserted into an analytical batch at a certain frequency (1 every 20 samples, for example). If samples from different projects and/or multiple collection points are assembled into the same analytical batch, the MS results reported for a particular project may be based on a spiked sample from a different project altogether. In this case, the MS information (pass or fail) may not provide any useable information to the data user.

Note that when analytical data is reviewed at the laboratory or verified/validated by a third party, the failure to recover matrix spikes within acceptance criteria is almost never a cause to reject data (except possibly for the specific sample that was spiked). Professional judgment plays more of a role when evaluating matrix spike results and when determining if sample results should be flagged as estimates. LCS, blank and other instrument quality control results are most often the drivers when qualifying data.

Customer Options Regarding Matrix Spikes

If project data quality objectives require matrix spike data be reported (whether for a specific sample of interest or to represent a group of samples), project officers should insure that these requirements are made clear to DLS through discussions with the Laboratory Consultants Office (LCO) and/or specifying the MS/MSD sample analysis requirements on the analytical request (document 330R).

Using the 330R request, extra sample jars/bottles (for the MS/MSD) should be ordered and the MS and MSD samples should be included in the final count of field samples. When this is done, the analysis of the MS and MSD samples will be charged at the same rate as the field samples.

If analysis of project-specific matrix spikes is not requested, and MS/MSD aliquots are not submitted, the samples used for matrix spiking (if the analytical method requires matrix spikes) will be selected at random at the prescribed batch frequency, and there may be no matrix spikes performed on samples specific to one or more projects in the batch. In this case, there will be no charge to the customer for analysis of the matrix spike samples.